

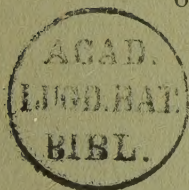
THE COEFFICIENT OF EXPANSION OF
NICKEL NEAR ITS CRITICAL
TEMPERATURE

BY

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A THESIS

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COEFFICIENT OF EXPANSION OF NICKEL NEAR ITS CRITICAL TEMPERATURE.

BY WALTER F. COLBY.

THE coefficient of expansion of nickel at ordinary temperatures was investigated by Fizeau in 1869. He used an interference method in which the expansion of the object was measured by the movement of a set of interference fringes. His method¹ of determining this displacement made possible the measurement of an expansion in the object under examination, of .02 wave-length of the light used. The delicacy of measurement was such that the coefficient of expansion of objects of but 1 cm. length could be determined with a high degree of accuracy. The improvements which have been made upon the method of Fizeau, notably by Benoit,² Abbe³ and Pulfrich,⁴ have been largely in the direction of increasing the ease and accuracy with which the displacement of the interference fringes can be observed and measured. In the Pulfrich interferometer, the instrument used in this investigation, we have undoubtedly the highest type of apparatus used for this purpose. In certain other methods which have been employed it has been necessary to use specimens 50 cm. long to reduce the percentage of error in measuring changes of length due to expansion. This greatly increases the difficulty in establishing and maintaining a uniform temperature, and in consequence only values of the mean coefficient over long ranges of temperature have been possible. In the present study it was desired so to refine the temperature measurements that ranges of five degrees might be used through a special region, *i. e.*, that of the critical temperature. The Pulfrich interferometer is extremely well adapted to this, allowing, as it does, high accuracy of measurements with small specimens.

¹ Ann. d. Chemie de Phys., 2, p. 143, 1864; 8, p. 335, 1866.

² Trav. et Mem. du Bureau Int. Poids et Mes., I., 1181; VI., 1888

³ Wied. Ann., 38, p. 453, 1889.

⁴ Zeitschr. für Instr., 1893, pp. 365, 401, 435.

Le Chatelier in 1888 published values of this coefficient for high temperatures, but not until recently were any precise measurements made in the critical region.

In 1901, Holborn and Day¹ studied the expansion of metal rods, 50 cm. long in an electrically heated porcelain tube. Readings were taken on scratches near the ends of the rods and the temperatures measured by means of a thermal couple placed at the middle. The results below 300° were expressed as a quadratic function of the temperature. Above 300° this function no longer held, the linear term becoming smaller and the quadratic larger. The temperature throughout the bar was far from uniform, and the method therefore could throw no light on the nature of the change at 300°.

Harrison² in 1904 made a similar investigation, using a nickel wire 60 cm. long, kept under tension by an adjustable spring. Lengths were measured by telescopes 10 cm. apart at the mid portion of the wire. He found an anomalous expansion at 370°, the critical temperature of his specimen. Between this temperature and 380° the wire experienced an elongation of about .05 per cent., whereas in the preceding ten degrees the elongation had been only .002 per cent. Above 380° the rate became .0019 per cent. per 10°. The discrepancy between these results and those of Holborn and Day may be explained by assuming that the elongation of the wire, due to the decrease of the elastic constant with rising temperature³ increased the absolute value of the coefficient. Temperatures were in this instance measured by the resistance of 20 cm. of the nickel wire itself, the calibration of which is not described in the article. The temperature recorded was therefore the mean throughout this length. The wire was placed centrally in a groove cut in a mahogany board and covered with a strip of wood with mica windows. Losses by unequal radiation and conduction through the leads were evidently considered too great for uniformity of temperature as results were computed only for fifty degree ranges.

Randall⁴ in a recent paper has described his work in this field. He worked with a solid nickel cylinder 1 cm. long and 2 cm. in

¹ Ann. d. Phys., 4, 104.

² Phil. Mag., June, 1904.

³ Pogg. Ann., 145, p. 147, 1872.

⁴ PHYS. REV., February, 1905.

diameter, employing an interference method and using the Pulfrich interferometer to measure the displacement of the fringe system. Temperature measurements were made with mercury thermometers, and although uniformity of temperature seemed certain, stem corrections may easily have introduced errors of such magnitude that measurement of temperature differences for intervals of less than twenty degrees became doubtful. With small ranges a slight constant error in each of the two temperatures, whose difference constituted the temperature interval, would not have materially altered the results. These stem corrections however were dependent on the temperature gradient of the oven packing, which in turn depended on the thermal history both of the oven and the room. Thus no error could be considered constant. Even these small ranges of twenty degrees may mask an anomaly. This specimen lost its magnetism at about 310° . The results showed no such anomaly at the critical point as those of Harrison. The coefficient increased steadily till about 275° and from there to 400° remained almost constant.

The present work grew out of this disagreement and takes as its problem the further refinement of temperature measurements and the investigation of other specimens of nickel. Three specimens have been used, which for convenience will be designated by the letters A, B and C. The work was begun in 1907 and preliminary tests made with specimen A. These were repeated in 1908 with slight modifications, and the same tests applied to B and C.

MAGNETIC MEASUREMENTS.

The first part of the problem consisted in finding definitely the temperature region in which magnetizability disappeared for the different specimens. A and B, being annular in shape, were wound ringwise with a primary and secondary of copper wire insulated with asbestos paste. A thermal junction of iron constantan encircled the nickel in direct contact with it, and was firmly fixed by the insulation. The whole was placed in an electric oven having an inner chamber of porcelain surrounded by 10 cm. of mineral wool. A current of one ampere was passed through the primary and the effect of its reversal noted by a ballistic galvanometer in

series with the secondary. Temperatures were measured by means of a Siemens and Halske potentiometer after the design of Lindeck and Rothe.¹ The preliminary tests were made first with temperature steadily rising to points somewhat above critical temperature and then steadily falling to room temperature, to ascertain the type of the curve. This excluded possibilities of overlooking an abrupt anomaly which might easily fall between two stationary temperatures. It gave, of course, no truly corresponding values of temperature and deflection, and, plotting these quantities as coördinates, the resulting curves for heating and cooling naturally included a considerable area, since the change of temperature of the interior of the nickel lagged behind that of the thermal junction. In the final tests each temperature was maintained from twenty to thirty minutes, allowing, it was thought, ample time for such thermal equilibrium as this test demanded. If the change of temperature was not great, the galvanometer deflection gradually increased during the first five minutes of the new temperature but was afterwards constant. Even in the steep portions of the curve the deflections gave no evidence of an unstable state. Although the included area was here greatly reduced, it did not disappear, showing a slight temperature hysteresis.

Fig. 3 (p. 518) shows the results obtained with specimen A. The loss of magnetic properties occurred here in the region between 370° and 380° and at temperatures just below 370° a decided rise in permeability was found both on heating and cooling. No such maximum occurred in the curve for specimen B (Fig. 4) although the method and strength of field were the same. Here the phenomenon took place at a very much lower temperature, between 280° and 310° , showing a marked difference in purity or in structure of the nickel. Specimen C was a cylinder 8 mm. in length and 2 cm. in diameter. The ballistic method was again used but the cylinder was placed inside a glass tube on which a primary and secondary were wound for a distance of about 4 cm. The sensitiveness was so much reduced that it was necessary to use a current of six amperes to obtain reliable results. Except in magnitude, the curve (Fig. 5) did not differ from that of specimen B, the region centering about 312° .

¹ Zeitschr. für Instr., 20, p. 285, 1900.

OPTICAL SYSTEM.

Since the method used in this investigation for measuring the changes in length caused by expansion, has been fully described,¹ it will be necessary only to outline it briefly and to denote adaptations to this problem. It consists fundamentally in the production of interference fringes by two plane reflecting surfaces, opposite and slightly inclined to one another, whose distance apart changes with the length of the specimen. The bands are viewed and their displacement measured by means of a Pulfrich interferometer (Fig. 1). Light from the source of illumination is focused on a totally

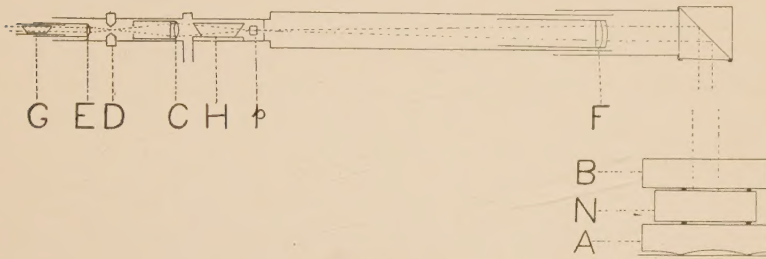


Fig. 1.

reflecting prism *p*, placed at the focus of the objective *F*. Approximately parallel rays emerge from the objective and are reflected downwards by another totally reflecting prism at the end of the interferometer tube. These rays are reflected from the optical surfaces, reënter the objective and after passing the free half of the telescope at prism *p*, and a second lens *C*, are brought to a focus at an adjustable horizontal slit *D*, immediately in front of two vertical cross hairs. Adjustment of lenses *C* and *E* brings the cross hairs and the images of the fringes in sharp outline. These are viewed through the direct vision spectroscope *G* and the horizontal images of the slit thus appear in the spectral colors. The images of the interference bands are visible in each of the slit images and can be made vertical by rotating the Dove's prism *H*. A micrometer attachment makes it possible to move the cross hairs parallel to themselves and thus to measure the distance of any interference band from a fiducial mark in the field.

¹ Pulfrich, *Zeitschr. für Instr.*, 1893, pp. 365, 401, 435. Randall, *Phys. Rev.*, February, 1905.

Specimen A was in the form of a ring 8.2 mm. long, 5 cm. internal diameter and 4 cm. external diameter. Specimen B, also a ring, was 2.3 cm. long, 2 cm. internal diameter and 3 cm. external diameter. Both ends of these rings had been ground away so as to form three equidistant feet. The optical system for these tests was formed by placing the nickel ring *N* on a quartz bed plate *A*, with upper surface plane polished and lower surface ground that it might give no disturbing reflections. The ring was surmounted by a similar plate *B*, both sides of which were plane polished. On the lower side was etched a small circle which was used as a point of reference in the measurements. To avoid reflections from the upper surface this cover plate was ground slightly wedge-shaped. For the preliminary tests this lower surface of the upper plate and the upper surface of the lower plate were used as reflecting surfaces, but their distance was so great that the clearness of the bands was not sufficient for the best results. For the final tests, accordingly, this air layer was reduced to a thickness of approximately 1 mm. by placing a fused quartz cylinder, with upper surface plane polished and lower surface ground so as to form three feet, on the bed plate *A* (Fig. 1) inside the nickel ring *N*. The coefficient of expansion of the quartz is so small in comparison with that of the nickel that when added to it it does not affect the first two significant figures. Any error in the value of the quartz coefficient or in the length of the quartz specimen would therefore appear outside the limits of error of the nickel. The increased distinctness of the bands doubtless more than compensated for the error introduced by this new material. Specimen *C*, being a circular cylinder, 1 cm. long and 2 cm. in diameter, was ground similarly to the quartz cylinder just described and placed inside a fused quartz ring. Since the surface of the nickel did not preserve its polish at high temperatures, it was surmounted by a thin fused quartz disk. This arrangement left an air layer of 0.2 mm. between the bottom of the cover plate and the upper surface of the quartz disk, producing a very satisfactory fringe system.

ILLUMINATION.

Illumination was produced by an Arons mercury lamp. The light was very brilliant and steady, and for the thin air layers used

in these tests, the green ($\lambda = 546 \mu\mu$) and two yellow bands ($\lambda_1 = 578.8 \mu\mu$, $\lambda_2 = 576.8 \mu\mu$) were sharply defined. These two yellow lines of the spectrum were not completely separated by the direct vision spectroscop of the interferometer but overlapped for a considerable part of their width. Consequently the dark interference bands which crossed them transversely were at times so completely superposed that settings were made on their common dark portion and the mean wave-length used in computations. Later in the same series the relative shift of these dark bands made it necessary to use the "upper yellow" alone.

THE OVEN.

The oven (Fig. 2) consisted of a vertical cylindrical iron frame formed by two rings 15 cm. in diameter connected by rods 35 cm. long. This, being well insulated with asbestos, was wound lengthwise with german silver wire *R* and surrounded by packing 8 cm. thick of alternate layers of asbestos and infusorial earth. This shell rested on a thick pad of asbestos through the center of which rose two porcelain rods, supports for the optical system. The top consisted likewise of an asbestos pad resting on a porcelain lid. An asbestos chimney *A*, fitted transversely with four squares of plate glass, penetrated the cover. All joints were closely packed with infusorial earth. The whole rested on a platform supported from the asphalt floor by steel rods clamped to the brick pier. The porcelain rods *P*, entering from below, supported a cylindrical brass chamber *C*, coated with asbestos and fitted at the top with a glass window. A brass disk *D*, resting on three leveling screws in the bottom, formed a base for the optical system. Two platinum thermometers *T* were firmly attached to the porcelain rods. The bulb of one was introduced into the inner chamber and the other placed in the air chamber outside in the same horizontal plane as the optical system. Since the heating coil was suspended in the outer chamber and free to air currents, it seemed evident that the temperature of that chamber would be almost immediately affected by any adjustment of the heating current, whereas the inner chamber because of its thermal capacity and asbestos coating would lag behind those changes and tend to remain constant, especially if the fluctuations

in the temperature of the surrounding air were somewhat rapid. The winding of the heating coil was such that it produced very slight effect on the magnetic field at the center of the oven. A

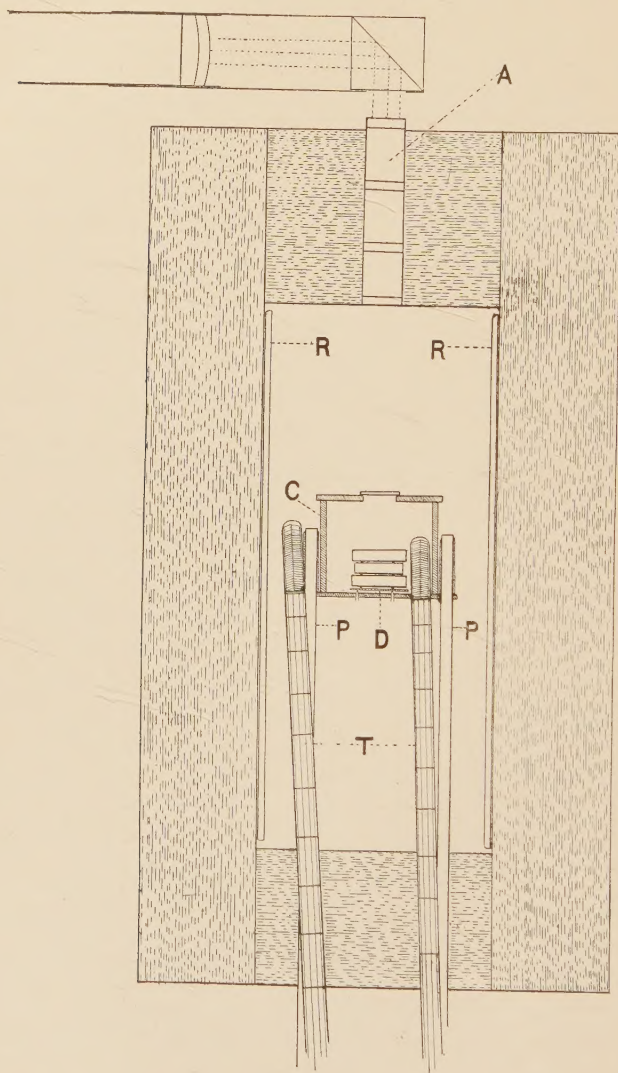


Fig. 2.

part of the heating current, however, was passed through a coil wound about the outside of the oven and so adjusted that no meas-

urable magnetic effect could be detected in the vicinity of the specimen.

THERMOMETERS.

The two thermometers were made by winding on mica vanes, fine platinum wires and gold soldering the terminals to heavy platinum leads, according to the description given by Callendar.¹ To eliminate the resistance of these leads a loop of similar wire identical in length was mounted close and parallel to them. The whole was fitted in a glass tube with the bulb end sealed. Circular mica disks through which the four leads passed were fitted at frequent intervals in the stem both to hold the leads in position and prevent air currents. The resistances of the thermometers were measured by means of a Carey-Foster slide wire bridge, the thermometer being used as an extension of the wire on one side and a resistance box and the dummy leads on the other. The lead resistance was thus automatically eliminated. The box and bridge wire were calibrated to six decimal places by comparison with standard resistances in the laboratory. One millimeter of the wire had a resistance of .000718 ohm and readings could be estimated to tenths of millimeters. The thermometers were calibrated by measuring their resistances in ice, steam, naphthaline vapor and sulphur vapor baths. The results were expressed by means of the parabolic formula given by Callendar, viz.,

$$t = 100 \frac{R_t - R_0}{R_{100} - R_0} + d \left(\frac{t}{100} - 1 \right) \frac{t}{100}.$$

R_0 for thermometer I., 39.59842 ohms.

d " " I., 1.554

R " " II., 40.82634

d " " II., 1.568

These values give a change of approximately one ohm for each ten degrees or .007° for each millimeter on the bridge wire. The galvanometer used was of such sensibility that the current passing through the bridge could be made small enough to eliminate heating effect in the thermometer. To guard against thermal effects

¹ Phil. Mag., 47, 191.

the wire and contact key of the bridge were of manganin, as were all resistance coils so that the entire circuit, with the exception of the platinum thermometers, was composed of manganin and copper. The junctions of the platinum leads with the copper leads connecting the thermometer with the bridge, were placed near one another to insure, if possible, the same temperature for all the junctions. These measures seem to have been sufficiently effectual, since no disturbing irregularities were experienced during the progress of the work.

SPHEROMETER.

A spherometer was used to measure the length of the rings and cylinders. It was made by the Geneva Society and read directly to .001 mm.

METHOD OF PROCEDURE.

To produce satisfactory fringes the lower surface of the cover plate and the upper surface of the bed plate must be inclined to one another at the proper angle. This angle was found by trial. The nickel rings were so adjusted by careful grindings of their feet, that, when placed between the cover and bed plates, fringes of the desired width were obtained. A fringe width of approximately one hundred scale divisions, as measured by the micrometer adjustment at the eye piece of the interferometer, was found to give most satisfactory results. It was feared there might be some irregularities due to air films or particles of dust between the points of contact of plates and rings. After the optical system had once been heated to 500° the band width was practically constant and without tilt, and on examination it even required some pressure of the fingers to separate the parts; moreover, while the application of pressure to the cover plate before heating produced a noticeable movement of the fringes, after being heated to 500° , a weight of 100 to 200 gms. on the cover plate produced no measurable effect. The oven was therefore heated to 500° before beginning each series, that this apparently more permanent condition might be attained.

The optical arrangements being thus in order, the oven was raised to the desired temperature by frequent adjustment of the heating current. To accomplish this the bridge was connected with the thermometer in the outer chamber and set at the desired reading,

the galvanometer being kept at the zero point by manipulation of a variable resistance in the heating circuit. For the first reading this was continued from three to five hours, for the later readings from two to three hours. Maximum variation in the outer chamber was $.04^\circ$, in the inner chamber about $.01^\circ$. The temperature of the inner chamber was recorded both before and after and sometimes in the midst of the reading taken on the fringe system with the interferometer. The temperature of the outer chamber was then changed and the number of bands passing the reference circle noted. Readings were taken on the green and yellow bands and Abbe's¹ computation used as a check on the number of bands counted during the shift. If δ_2 be the distance from the zero point to the nearest band in fractions of a band width at the higher temperature, δ_1 a similar quantity at the lower temperature and M the whole number of bands passed, then the apparent shift $t_1 = M + \delta_2 - \delta_1$.

At each interferometer reading a barometer reading was also taken and Pulfrich's² correction applied for change of wave-length in the air layer due to change in density. This correction is

$$K = d(t_2 - t_1) \frac{b_1}{760} \frac{1}{1 + \alpha t_1} \frac{1}{1 + \alpha t_2} \left[\frac{2(N - 1)\alpha}{\lambda} \right] \\ - d(b_2 - b_1) \frac{1}{1 + \alpha t_2} \left[2 \frac{N - 1}{\lambda} \frac{1}{760} \right].$$

Where d is the thickness of air layer, t_2 the higher temperature, t_1 the lower temperature, b_2 and b_1 the corresponding barometric readings, α the coefficient of expansion of air and n the index of refraction of air under standard conditions. K is here expressed in terms of band widths. The first term, a correction for the increased temperature, is always positive. The second, a correction for change in barometric pressure, is negative when $(b_2 - b_1)$ is positive, that is, when the greater pressure occurs at the higher temperature. If the apparent shift t_1 be taken as positive for increasing and negative for decreasing thickness of air film, the absolute value of true shift $f = f_1 + k$. The change of length is $f \cdot \lambda / 2$.

¹ Wied. Ann., 38, 1889, p. 473.

² Zeitschr. für Instr., p. 437, 1893.

DEGREE OF ACCURACY.

The formula expressing the value of the coefficient of expansion is

$$\alpha = \frac{f \frac{\lambda}{2}}{L(t_2 - t_1)} + \frac{la'}{L}.$$

Where l and a' denote respectively the length and coefficient of expansion of the fused quartz cylinder or ring and L the length of the nickel specimen. The value of a' was taken from recent, unpublished work of Randall's in which he expresses the true coefficient by the parabolic formula:

$$\alpha \cdot 10^8 = 41.94 + .156\theta - .000225\theta^2,$$

giving an almost constant value through this region of 68×10^{-8} .

The value of f , the number of displaced fringes, is dependent on two measurements each of which can be determined within a probable error of .01 band, giving a probable error for f of $\pm .015$. The temperature interval $(t_2 - t_1)$ is likewise dependent on two measurements. The sensitiveness of the bridge made readings of the platinum thermometers definite to half millimeters on the bridge wire or $.0035^\circ$. Variations in these readings, however, might be due to two causes, actual variation in the inner chamber and irregularities of the bridge. Two or three readings were taken for each determination of the band position, these determinations occupying approximately fifteen minutes. These temperature readings were usually very much in accord, and in fully seven eighths of the observations did not vary more than $.01^\circ$. In a few cases, irregularities in the heating current which could not be properly compensated during the band determination, caused fluctuations in the inner chamber as great as $.07^\circ$. For the greatest of these variations, the temperature was maintained an hour longer and the reading repeated. The probable error for the majority of the temperature readings was computed from the sets of temperature readings taken during a determination of the band position and found to be $\pm .0075^\circ$, giving for $(t_2 - t_1)$ a probable error of $\pm .010^\circ$. The length of the nickel L may be determined within a mean error of $\pm .001$ mm.

For specimen A the shift for a change of temperature of ten degrees was about five bands. The error in α due to f for this interval amounts to 4.9×10^{-8} ; due to error in L , $.2 \times 10^{-8}$; due to error in $(t_2 - t_1)$, 1.6×10^{-8} ; giving a probable error in α due to the three quantities of $\pm 10.2 \times 10^{-8}$. Similarly for specimen B. Its length was 23.01 mm. and the shift for ten degrees, 14 bands. The error in α for 10° intervals was $\pm 2.4 \times 10^{-8}$, for 5° intervals $\pm 4.8 \times 10^{-8}$. Specimen C was 7.88 mm. long and gave for 10° a shift of 5 bands. 10° intervals, accordingly, gave results within a probable error of $\pm 5.45 \times 10^{-8}$ and 5° intervals $\pm 10.9 \times 10^{-8}$.

In series II. of specimen A, two successive values of α lie considerably outside this probable error. It was not possible to trace the cause of this but since the first is too high and the second too low, it

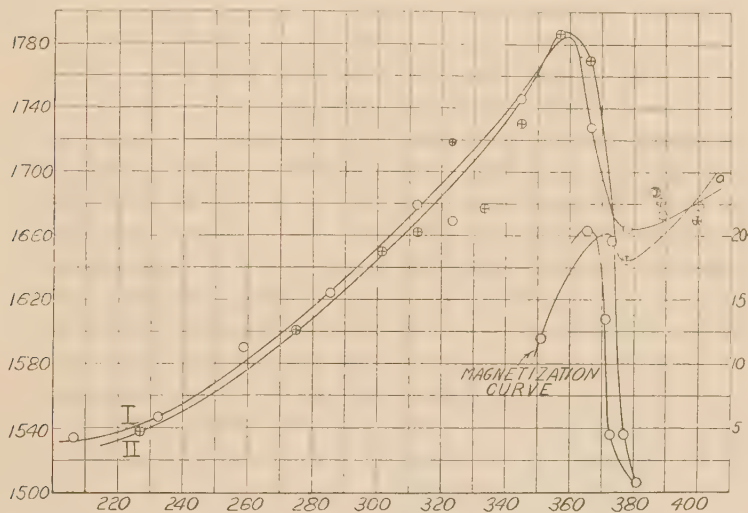


Fig. 3. Specimen A.

does not seem unreasonable that a mistake was made in reading the mid temperature on which they both depended. It was possible to pass a consistent curve through the majority of the values of α . In fact only six of the results were distant from these curves by an amount exceeding their respective mean errors.

The results for the three specimens are shown in Figures 3, 4, and 5. During the first series, with specimen C, corrosion of

the bridge wire so reduced the sensitiveness of the temperature measurements that all results for less than 20-degree intervals were discarded. A thorough cleaning of the bridge restored accuracy for series II. As had been found in the preliminary tests, series II. could not be combined with series I., the value of the coefficient being dependent on the thermal history of the specimen. The values of the coefficient have been plotted as ordinates, with mean temperatures as abscissas, and the resulting curves show a striking similarity for the three specimens.

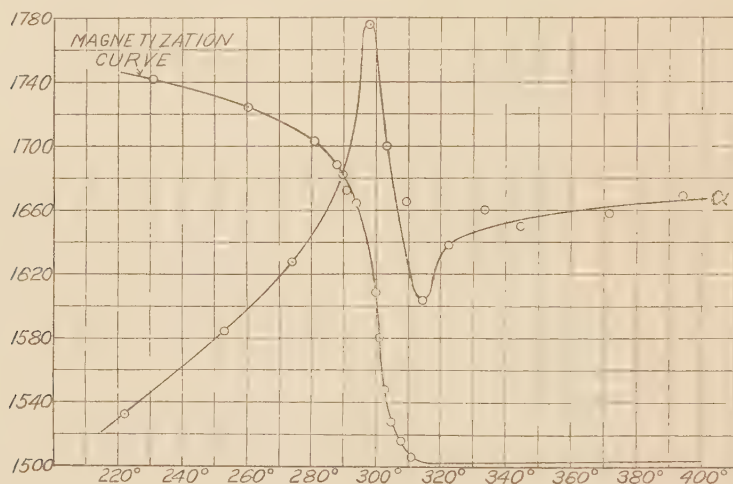


Fig. 4. Specimen B.

Specimen A is electrolytically prepared, and although a quantitative analysis has not yet been secured, it may be presumed to be unusually free from impurities. A qualitative test shows only a trace of cobalt. Its critical point lies 60° higher than the others and coincides very closely with the anomaly in expansion. This coincidence is equally good for the other two specimens, although the anomaly itself varies in magnitude. Specimen B was cut from a rolled nickel rod obtained from the Westphalian Nickel Works, and judging from its color on oxidation, contains far more iron than A and doubtless other impurities. Specimen C is the one used by Randall and was prepared by Zeiss of Jena. Its color on oxidation is similar to that of B, but the anomaly is far less marked.

For the sake of comparison with Randall's curve, computations of α were made for 20-degree intervals and the resulting curve plotted. With one or two exceptions, the values did not vary from his outside the limits of error.

No results have been given from the preliminary work with specimen A in 1907, but at least a brief mention of them should be made. The oven and optical system in this series differed in no way from those of 1908, but instead of the platinum thermometers, two groups of seven copper advance thermal junctions were used. One group was placed at equal intervals about the nickel in the inner chamber and the other similarly in the outer chamber. A Wolff potentiometer was used to measure their E.M.F. Although their sensitiveness and quick response was very satisfactory, the copper

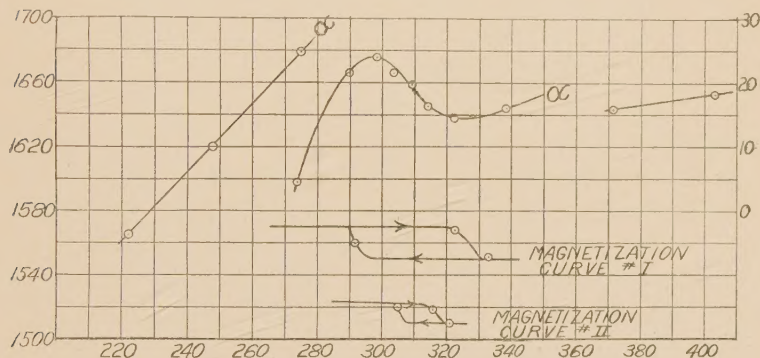


Fig. 5. Specimen C.

oxidized so readily at the high temperature and they soon became so fragile, that it was impossible to work with them. The observations in this work were unfortunately always discontinued at the close of each day and the isolated groups of about five readings, although consistent with each other, could never be satisfactorily combined with those of the following day. As mentioned above, this was due to the well-known change in the value of the coefficient of expansion of metals at a given temperature after the metal has been heated. The type of the curves which the readings suggested, however, agreed with that found in the later work, showing a decrease in a special region and an increase on either side.

In conclusion the results may be summarized as follows:

1. The electrolytically prepared specimen shows a high value of permeability just below its critical temperature. This phenomenon is absent in the other specimens and is possibly due to the greater purity or crystalline structure of specimen A.

2. For the first sample, the critical temperature is in the neighborhood of 370° and, as in the case of iron, is lowered by impurities.

3. The absolute value of the expansion coefficient is in part dependent on the history of the specimen.

4. Through a continuous heating the relative values show an increase at the beginning of the region followed by a decrease.

5. Before and after the critical regions, the coefficient increases at a regular rate.

The writer wishes to express his gratitude to Professor Randall both for his aid in the final determinations and his invaluable suggestions throughout the work.

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